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EFFECTS OF LOW-PRESSURE AIR ON OXYGEN CONTAMINATION AND LITHIUM CORROSION OF A TANTALUM ALLOY, T-111, AT 980° AND 1260° C

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16. Abstract

A study was made to determine the effects of low-pressure air on contamination and corrosion in the tantalum alloy T-111/lithium system at 980° and 1260° C. Capsules of T-111 containing lithium were exposed to six vacuum levels between 1×10^{-8} and 3×10^{-4} torr by controlled air leakage into a vacuum system. Capsules exposed at 980° C and 2×10^{-4} torr failed from intragranular oxidation. The remainder of the capsules completed the 96-hour tests. The depth of oxygen contamination was greater at 980° C than at 1260° C. Tests made at 10^{-4} and 10^{-5} torr levels caused large increases in the oxygen content of the T-111. Tests at 10^{-6} torr or less produced no significant contamination. No lithium corrosion of the T-111 was observed under any of the conditions.

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EFFECTS OF LOW-PRESSURE AIR ON OXYGEN CONTAMINATION AND LITHIUM CORROSION OF A TANTALUM ALLOY, T-111, AT 980° AND 1260° C

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SUMMARY

Capsules of the tantalum alloy T-111 (Ta-8W-2Hf) containing lithium were heated under various degrees of vacuum to simulate the environment for ground testing of large prototype space nuclear electric power systems. The capsules were tested at 980° and 1260° C between pressures of 1×10^{-8} and 3×10^{-4} torr for 96 hours. The desired vacuum level was obtained by using a controlled leak in the system to simulate leaks which are encountered in large vacuum systems. The effect of thermal cycling was also determined.

Capsules tested at 980° C and 10^{-4} torr failed at the weld after about 24 hours from air contamination. Other capsules tested at 980° C between 1×10^{-8} and 2×10^{-5} torr completed the 96-hour tests.

All capsules tested at 1260° C between the pressures of 1×10^{-8} and 2×10^{-4} torr completed the 96-hour tests.

Capsules tested at 10^{-4} torr exhibited severe internal oxidation extending inward from the outer wall. There was no significant pickup of interstitials other than oxygen. The inside, or lithium side, of the capsules was unaffected. The lithium in these capsules showed significantly increased oxygen contents, indicating that oxygen had diffused completely through the wall. Oxides recovered from the T-111 indicated that at 980° C oxygen reacted primarily to form tantalum oxides. However, at 1260° C the oxygen reacted primarily to form hafnium oxides.

Capsules tested at 2×10^{-5} torr showed oxygen contamination to depths of about 0.5 and 0.4 millimeter in 96 hours at 980° and 1260° C, respectively. At pressures of 2×10^{-6} torr or less, no oxygen contamination was detected.

Thermal cycling between the test temperature and 540° C did not affect the system. It is concluded that T-111 space power system components could be ground tested at air pressures of 2×10^{-6} torr for as long as 100 hours, but pressures of less than 2×10^{-7} torr would be required for longer term tests.

INTRODUCTION

A large nuclear space power system which has been under study by NASA would employ a liquid alkali metal such as lithium as the thermodynamic working fluid and fabricable refractory metals such as tantalum for the majority of those system components in contact with the working fluid. The tantalum alloy T-111 (Ta-8W-2Hf) is favored for these applications because of its combination of desirable mechanical properties and resistance to corrosion by lithium. The corrosion resistance of both tantalum and T-111, however, is known to decrease with increasing oxygen content of the metal. Tantalum and its alloys are also well known for their ability to absorb oxygen in poor vacua at elevated temperatures, causing a change in mechanical properties. The development of space power systems will involve ground testing of large segments of the systems in large vacuum chambers such as the NASA Plum Brook Space Power Facility which, because of its size, presently cannot be maintained at less than about 10^{-6} torr. In view of the possibility of gaseous contamination and the attendant decrease in resistance to lithium corrosion and change in mechanical properties of T-111 during ground testing, it is important to determine the tolerable vacuum level for long-time containment of lithium by T-111.

The corrosion of oxygen-doped tantalum by lithium has previously been studied by Klueh (ref. 1) and by Stecura (ref. 2). The threshold oxygen concentrations for grain boundary attack were determined as 100 ppm by weight at 600° C and between 20 and 170 ppm by weight at 1115° to 1390° C. Increasing the oxygen content from these threshold values to 800 ppm progressively increased the extent of lithium corrosion. Similar observations have been made on the corrosion of oxygen-doped niobium by lithium (refs. 1, 3, and 4).

The effects of oxygen doping and heat treatment on the lithium corrosion of T-111 at 500° and 1000° C have been studied by Sessions and DeVan (ref. 5). Although cold-worked T-111 containing 100-ppm oxygen was unattacked at 1000° C, increasing the oxygen content to 1500 to 2500 ppm promoted severe corrosion at both 500° and 1000° C. Corrosion of T-111 containing as much as 2500-ppm oxygen, however, was eliminated by a prior heat treatment at 1300° C. This heat treatment allowed reaction of the oxygen with hafnium in the T-111 to form a precipitate of hafnium oxide (HfO_2), considerably reducing the amount of oxygen dissolved in the alloy matrix and grain boundaries and thus reducing the attack by lithium.

The present study was conducted to determine the effect of low air pressures on the contamination of T-111 and the accompanying possible effects on the rate of corrosion. This study differed from previous ones since contamination of the T-111 was occurring while the T-111 was in contact with the lithium. This approach is

representative of actual reactor ground testing conditions. Capsules of T-111 with a 1-millimeter-thick wall were filled with lithium and heated for as long as 96 hours at 980° and 1260° C in a high-vacuum system. Pressures were varied between 10^{-8} and 3×10^{-4} torr by controlled air leakage into the system. The effect of thermal cycling between the test temperature and 540° C under the same experimental conditions was also studied to simulate operation of a space power system. The extents of contamination and corrosion were evaluated by metallographic examinations and chemical analyses.

EXPERIMENTAL APPARATUS AND PROCEDURE

Lithium-Filled T-111 Capsules

Capsules of T-111, 4.4 centimeters long by 1.3 centimeters in diameter by 0.10 centimeter in wall thickness (1.75 in. by 0.5 in. by 0.040 in.), were machined from cold-worked bar stock. The capsules were cleaned and given a 1-hour 1316° C heat treatment prior to lithium filling. About 1 gram of lithium was put into each capsule in a vacuum chamber. A tantalum wire, 3.81 centimeters by 0.051 centimeter in diameter (1.5 in. by 0.020 in.) was placed inside each capsule to ensure flow of liquid lithium to the bottom of the capsule. The capsules were sealed by electron beam welding. Two platinum/platinum-rhodium thermocouples were then attached to the capsule wall. The capsule assembly is shown in figure 1.

Apparatus

A schematic diagram of the vacuum system used for testing the capsules is shown in figure 2. The system consisted of an ion pump, a liquid-nitrogen-trapped diffusion pump, an induction coil, a variable leak valve, and a pressure gage.

The ion pump was used to obtain as low a pressure as possible for control tests. The cold-trapped diffusion pump was used for testing at the higher pressure levels. Increased pressure levels were obtained by bleeding air into the system through the controlled leak valve. A constant dynamic pressure was maintained throughout each run.

The capsule was heated by an induction coil powered by a 5-kilowatt radiofrequency generator. The glass furnace section was cooled with an air blower. Pressure measurements were taken from an ionization gage located near the furnace section.

Test Procedure

Capsules were placed in the heater section and the system was evacuated. The capsules were then heated gradually to the test temperature, keeping the pressure less than 1×10^{-6} torr. After the pressure had decreased to 1×10^{-7} torr, the variable leak valve was opened until the system pressure was at the desired level. For isothermal tests, the temperature was held constant. For cyclic tests, the temperature was varied between the test temperature and 540° C eight times. The temperature was increased and decreased at the rate of 20° C per minute and was held at 540° C for 1 hour. Following each 96-hour test, the leak valve was closed, and the capsule was cooled to room temperature and removed from the system.

Post-Test Evaluation

The capsules were opened in an inert atmosphere and transferred in the distillation transfer chamber to the vacuum distillation apparatus described in references 6 and 7. The lithium was removed by vacuum distillation at about 730° C for 2 hours and a pressure of 1×10^{-5} torr.

Following distillation, the remaining residue was analyzed for oxygen content by acidimetry, and the capsule material was prepared for chemical and metallographic examination. Chemical analyses were performed for oxygen, nitrogen, carbon, and hydrogen. Metallographic specimens were examined for lithium penetration, variations in microstructure, and the presence of precipitates. Knoop microhardness traverses were made on cross sections of the capsule walls.

RESULTS

General Observations

The experimental conditions and chemical analyses of the capsule materials and lithium are given in tables I and II. Microstructures of the T-111 are shown in figures 3 to 12. Hardness traverses of the wall section of the capsules are shown in figures 13 and 14.

Sixteen of the capsules completed the 96-hour test period. However, both capsules tested at 980° C and 10^{-4} torr failed after about 24 hours when lithium began leaking from the capsule.

Chemical Analysis of Capsule Materials

All control capsules run at 1×10^{-8} torr or less at 980° and 1260° C in the ion-pumped system showed no increase in interstitial content from the as-received material. The control capsules run at the ultimate diffusion pump pressure of 6×10^{-8} torr also showed no change at either temperature.

At 5×10^{-7} torr, capsules run at 980° C did not have a detectable increase in oxygen or nitrogen. At 1260° C, there was about a 10-ppm increase in oxygen but no change in nitrogen.

At 2×10^{-6} torr, oxygen concentration increased about 100 ppm at both temperatures, but nitrogen showed no significant increase.

At 980° C and 2×10^{-5} torr the oxygen content increased by about 520 ppm, while the nitrogen concentration remained unchanged. At 1260° C and 2×10^{-5} torr, the oxygen content was about 700 ppm and the nitrogen content increased by about 20 ppm.

At the highest pressures of 2×10^{-4} and 3×10^{-4} torr, the two capsules tested at 980° C picked up about 600-ppm and 700-ppm oxygen, respectively, prior to their failures at 24 and 18 hours. The nitrogen contents, however, decreased by about 15 ppm.

The two capsules tested at 1260° C and 2×10^{-4} torr completed the 96-hour tests. Both picked up about 3600-ppm oxygen. Nitrogen content increased about 30 ppm at 1260° C. The hydrogen and carbon changes were insignificant.

There seemed to be no effect of thermal cycling on the interstitial content of any of the capsules.

Portions of the capsules tested at 2×10^{-4} torr and 980° and 1260° C were dissolved in a bromine-methyl alcohol solution to extract the insoluble metal oxides. The extracted oxide particles were then analyzed by emission spectroscopy. Results of the analysis indicated the metal content of the residue from the capsule tested at 980° C to be almost completely tantalum with traces of hafnium and tungsten. Metal content of the residue recovered from the capsule tested at 1260° C was completely hafnium. An X-ray diffraction analysis of the extracted residues also indicated the 980° C-capsule residue was primarily a mixture of tantalum oxides and the 1260° C-capsule residue was HfO_2.

Chemical Analysis of Lithium for Oxygen

The residue remaining inside the capsule following lithium distillation was dissolved in water, and the solution was analyzed for alkalinity. Results of the titration were converted to oxygen content in the lithium assuming the oxide to be Li_3O_2 (ref. 8). The oxygen results are given in tables I and II.

In general, there were no significant differences among capsules tested at 10^{-8}, 10^{-7}, 10^{-6}, and 10^{-5} torr ranges. However, at 2×10^{-4} torr, increased oxygen content in the lithium was observed. At 980° C, an average concentration of about 900-ppm oxygen was found for the two capsules. Capsules run at 1260° C and 10^{-4} torr, however, had extremely high oxygen content. Oxide residue coated the walls, and there was a heavy buildup in the bottom of the capsules. Quantitative measurement on one of the capsules indicated approximately 53 000-ppm oxygen in the lithium. Quantitative measurement of the oxide in the other capsule was not possible since the lithium distillation was incomplete, probably because of the large amount of oxide.

Thermal cycling did not appear to affect the oxygen content of the lithium.

Metallography

A photomicrograph of the annealed starting material is shown in figure 3; it is equiaxed and single phase.

Capsules tested at 980° C and 2×10^{-4} and 3×10^{-4} torr failed in less than 24 hours from intergranular oxidation. Photomicrographs of the 3×10^{-4}-torr specimen are shown in figure 4. Severe oxygen contamination occurred at the outside wall of the capsule and was both inter- and transgranular. In the area of failure, the material was weak and grains pulled out of the matrix during handling. As-polished weld and wall specimens exhibited grain boundary staining upon standing overnight from the seapage of water picked up during polishing. Metallographic etching failed to reveal any further evidence of attack. Failure of these two capsules is attributed to oxygen contamination and grain boundary oxidation, since no evidence of lithium corrosion was seen at any area of the interior surface of the capsules.

Intergranular oxidation occurred to a depth of about 0.6 millimeter on the capsule tested at 3×10^{-4} torr under isothermal conditions and to a depth of about 0.1 millimeter on the material tested at 2×10^{-4} torr under cyclic conditions. The hardness profile for the material tested at 980° C and 2×10^{-4} torr is shown in figure 13. Hardness decreased almost linearly from the outside wall to the inside, indicating an oxygen concentration gradient across the wall. Hardness decreased to that of the control samples at the inner wall.

The wall cross section of a capsule tested at 980° C and 2×10^{-5} torr is shown in figure 5. About one-half of the 1-millimeter-thick wall appears to be affected by oxygen contamination. The harder material near the outer wall was more difficult to etch than the soft material near the inner wall, which made it possible to estimate the depth of oxygen penetration. The inner half of the wall was not hardened and exhibited

a microstructure similar to that of the control specimen. Hardness measurements, shown in figure 13, indicate that, at about 0.5 millimeter from the outside wall, hardness had decreased to the level of the 980° C control samples. This measurement corresponds to the apparent visible depth of oxygen contamination.

The wall cross section from capsules tested at 2×10^{-6} (fig. 6), 5×10^{-7} (fig. 7), and 1×10^{-8} torr (fig. 8) and 980° C were similar. These specimens showed the longitudinal streaks of unidentified grain boundary precipitate which are typical of T-111 aged at 980° C. Hardness profiles shown in figure 13 indicate no change in hardness from the outside to the inside of the 5×10^{-7}- and 1×10^{-8}-torr specimens. The hardness of the 2×10^{-6}-torr specimen was greatest at the outside surface and leveled off at a depth of about 0.25 millimeter.

All capsules tested at 1260° C completed the 96-hour exposures at the various pressures. Observation by optical microscopy of etched wall specimens from the capsule tested at 2×10^{-4} torr (fig. 9(a)) indicated the presence of two different structural regions across the wall. At the outer wall, both inter- and intragranular oxidation are evident. Oxidation at the outer surface occurred to a depth of about 0.4 to 0.5 millimeter on both isothermal and cycled T-111. Hardness measurements (fig. 14) also indicated a sudden change at this distance. Hardness in this instance increased from the outside to the inside. After polished specimens stood in air, the moisture trapped in the porous phase stained the surface, giving it an etched appearance. The inner portion of the capsule wall was difficult to etch, similar to the inner portion from the capsule tested at 980° C and 3×10^{-4} torr (fig. 4(a)). The high hardness and etching difficulty are attributed to saturation of the matrix with oxygen. An optical photomicrograph of the weld area is shown in figure 9(b). The weld also was oxidized intergranularly and appears to be near failure. The dark portion is the capsule lip. This area was surrounded by lithium and apparently consists of a heavy HfO_2 precipitate in a tantalum-tungsten matrix which is not saturated with oxygen.

Electron microscope examination across the wall confirmed the two types of microstructure observed by optical microscopy. Two-thirds of the inner wall (~0.6 mm) contained uniformly equiaxed grains. The remaining one-third (~0.4 mm) of the wall had fissures running perpendicular to the outer wall. Precipitates were observed, but only in the portion having the equiaxed grains. These precipitates were identified by X-ray diffraction as HfO_2.

A specimen tested at 1260° C and 2×10^{-5} torr is shown in figure 10. The depth of internal oxidation is indicated by the differences in the etching to be about 0.25 millimeter from the outside surface. The microstructure of the remainder of the wall was similar to that of the 1260° C control capsules. There was a decrease in hardness (fig. 14) from the outside wall to a depth of about 0.25 to 0.4 millimeter, corresponding

to the metallographic observations. Electron microscope examination across the wall indicated inter- and intragranular precipitates not observed by optical microscopy. Particles observed near the inside edge are the unidentified precipitate which occurs in all T-111 on aging in this temperature range.

At 2×10^{-6} (fig. 11), 5×10^{-7}, and 1×10^{-8} (fig. 12) torr and at both 980° and 1260° C, there appear to be no significant differences in the metallography of the T-111. Both materials exhibit the typical grain boundary precipitation observed in T-111 aged at these temperatures.

DISCUSSION

There are dramatic differences between the results of this study and those from previous studies on the effects of oxygen contamination on the corrosion of tantalum and tantalum alloys by lithium. In previous studies, corrosion was observed in tantalum containing less than 200-ppm oxygen and in T-111 containing 1500-ppm oxygen. However, in this study, no lithium corrosion was observed at oxygen concentrations as high as 3600 ppm in the T-111. This difference is attributed to the differences in oxygen content at the time of initial exposure of the alloy to lithium. If oxygen preexists at large concentrations either in solution or as tantalum oxides, rapid penetration of the metal by lithium occurs. However, if the oxygen preexists in the tantalum matrix at a low level, either by virtue of being tied up as HfO_2 or simply being present only at a low level (as in the present study), corrosion does not occur. Since lithium has such a high affinity for oxygen, the slow addition of oxygen to T-111 while it is exposed to lithium results only in gettering of oxygen from T-111 by lithium at the T-111/lithium interface. Presumably, as long as the rate of oxygen supply to the interface approximates the rate of oxygen removal by lithium, a low-oxygen T-111/lithium interface can be maintained and corrosion of T-111 by lithium will not occur.

Dynamic transfer of oxygen through T-111 to lithium was quite evident in the present study at pressures of 2×10^{-4} and 3×10^{-4} torr, where the oxygen content of the lithium increased substantially. Even though corrosion failure of the T-111 did not occur, this situation would be quite detrimental to operation of a T-111/lithium heat pipe or power system, since oxide plugging would restrict lithium flow at constrictions and in cooler sections.

The potential for failure of T-111/lithium systems by internal oxidation in poor vacua appears greater than the potential for failure by lithium corrosion. Metallography reveals that oxidation occurs in three stages, as can be seen in the micrographs of the capsule walls after exposure at 2×10^{-4} to 3×10^{-4} torr (figs. 4 and 9). The first

stage is oxidation of hafnium to HfO_2, which occurs to a considerably greater extent at 1260° C than at 980° C. The second stage is saturation of the tantalum-tungsten matrix with oxygen to form the hard, unetched zone visible after exposure to air pressures of 2×10^{-5} torr or higher. The third stage consists of actual intergranular oxidation and the formation of intragranular oxide platelets on preferred planes of the matrix. Areas containing those porous oxides are difficult to prepare metallographically because they easily stain. These areas are visible on the specimens exposed at 2×10^{-4} to 3×10^{-4} torr (figs. 4 and 9). The weakness of these regions is evident in figure 4, where a number of grains fell out during specimen preparation. Failure of the two capsules at 980° C and 2×10^{-4} to 3×10^{-4} torr is attributed to the weakness of these intergranularly oxidized regions in the thin areas of the welds.

The change in strength and ductility of T-111 due to oxygen contamination was investigated by Liu (ref. 9) and Etter and Smith (ref. 10) under experimental conditions similar to those of the present study. A specimen of T-111 which was oxygen contaminated at 1000° C without an additional heat treatment exhibited a much lower tolerance for oxygen contamination than those specimens which received a 1700° C heat treatment. Without the additional heat treatment, there was a general increase of strength and decrease of ductility to oxygen contents of about 800 ppm. However, above 800 ppm, strength decreased gradually and ductility was lost completely. Contamination of T-111 with oxygen at 1400° C followed by testing at 1204° C indicated that the ultimate tensile strength and yield strength were only slightly affected by oxygen contamination to 6000 ppm. Elongation was unaffected to 2400 ppm; however, it decreased steadily at higher levels, to 6000-ppm oxygen. Therefore, a system must be designed to compensate for loss of strength or the onset of brittleness that may be encountered because of oxygen contamination.

The results of this study indicate that T-111/lithium systems with a 1-millimeter-thick T-111 wall could be safely tested at a residual air pressure of 2×10^{-5} torr for as long as 96 hours. Under these conditions, moderate oxygen contamination of the T-111 will occur. However, it is recommended that systems be tested at lower pressures if conditions permit. For tests of longer duration, lower pressures will be required, the level depending on the length of the test. The relationship between time and pressure on contamination is given in reference 11. Essentially, a tenfold decrease in pressure is required for each tenfold increase in test time. Therefore, a pressure of 2×10^{-7} torr would be required for a 1000-hour test to maintain the contamination level produced at 2×10^{-6} torr in this study.

CONCLUSIONS

The following conclusions were drawn from this study on the effects of low-pressure air contamination on the corrosion of T-111 by lithium:

1. No detectable corrosion reaction occurs between T-111 and lithium for exposure times as long as 96 hours at 980° and 1260° C and residual air pressures from 1×10^{-8} to 3×10^{-4} torr.

2. The extent of oxygen contamination increases with increasing residual air pressure and can cause mechanical failure by intergranular oxidation. Slight nitrogen contamination occurs at 1260° C but is insignificant compared to oxygen contamination.

3. Oxygen contamination occurs to a greater depth at 980° C because of the inability of hafnium to getter oxygen from solution. At 1260° C, a gettering reaction does occur, substantially reducing the depth of oxygen contamination. This behavior is most evident at higher air pressures, such as 10^{-4} torr.

4. Slow cycling between 980° or 1260° C and 540° C does not affect the rate of oxygen contamination or of lithium corrosion as compared to isothermal heating at the same temperatures.

5. For satisfactory testing of space power systems the vacuum level required depends on the expected test time. Based on results of these tests, systems could be exposed for 100 hours at 10^{-5} torr with only moderate oxygen contamination of the T-111. However, to avoid contamination a pressure of the order of 10^{-6} torr is recommended. For times greater than 100 hours a decade decrease in pressure is recommended for each decade increase in time.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 25, 1973,
502-25.

REFERENCES

1. Klueh, R. L.: The Effect of Oxygen on the Corrosion on Niobium and Tantalum by Liquid Lithium. Rep. ORNL-TM-4069, Oak Ridge National Lab., Mar. 1973.

2. Stecura, S.: Corrosion of Oxygen-Doped Tantalum by Lithium. NASA TM X-2989, 1974.

3. Brehm, W. F., Jr.; Gregg, J. L.; and Li, Che-Yu: Grain Boundary Penetration of Niobium (Columbium) by Lithium. Trans. AIME, vol. 242, no. 7, July 1968, pp. 1205-1210.

4. Hoffman, E. E.: The Effect of Oxygen and Nitrogen on the Corrosion Resistance of Columbium to Lithium at Elevated Temperatures. Rep. ORNL-2675, Oak Ridge National Lab., Jan. 27, 1959.

5. Sessions, C. E.; and DeVan, J. H.: Effect of Oxygen, Heat Treatment, and Test Temperature on the Compatibility of Several Advanced Refractory Alloys with Lithium. Rep. ORNL-4430, Oak Ridge National Lab., Apr. 1971.

6. Gahn, Randall F.: Determination of Oxygen in Lithium by the Vacuum Distillation Method. NASA TN D-5197, 1969.

7. Gahn, Randall F.: Technique for Recovery of Alkali-Metal Reaction Products. NASA TN D-4310, 1968.

8. Stecura, Stephan: Identification of a New Lithium Oxide, Li_3O_2. NASA TM X-2888, 1973.

9. Liu, C. T.: Effect of Oxygen Contamination on the Mechanical Properties of T-111. Fuels and Materials Development Program Quarterly Progress Report for Period Ending December 31, 1971. Rep. ORNL-TM-3703, Oak Ridge National Lab., Mar. 1972.

10. Etter, D. E.; and Smith, W. H.: Effect of Oxygen Contamination on the Tensile Properties of T-111 (Tantalum - 8 Tungsten - 2 Hafnium) at 1204° C. J. Less Common Metals, vol. 27, 1972, pp. 109-122.

11. Rosenblum, Louis; Englund, David R., Jr.; Hall, Robert W.; Moss, Thomas A.; and Scheuermann, Coulson: Potassium Rankine System Materials Technology. Space Power Systems Advanced Technology Conference. NASA SP-131, 1966, pp. 169-199.

TABLE I. - ANALYSES OF T-111 AND DEPTH OF

CONTAMINATION AFTER EXPOSURE AT 980° C

System pressure, torr	Exposure time, hr	Analysis of T-111,[a] ppm				Depth of contamination,[b] mm	Oxygen content of lithium, ppm
		O	N	C	H		
---	0	36	30	22	0.6	---	110
Isothermal exposures							
[c] 1×10^{-8}	96	31	24	---	0.5	0	---
[d] 6×10^{-8}	↓	32	24	26	<.5	0	190
5×10^{-7}		37	32	---	---	0	---
2×10^{-6}		129	36	---	---	.05	190
2×10^{-5}		563	34	---	<.5	.8	230
3×10^{-4}	[e] 18	630	13	---	2.5	[f] 1.0	990
Cyclic exposure between 980° and 540° C[g]							
[c] 1×10^{-8}	96	37	21	---	<0.5	0	90
2×10^{-5}	96	543	30	---	< .5	.5	200
2×10^{-4}	[e] 24	711	17	---	.9	[f] 1.0	1300

[a] Oxygen and nitrogen determined by vacuum or inert-gas fusion. Carbon determined by combustion. Hydrogen determined by vacuum extraction.

[b] Determined by optical metallography and microhardness.

[c] Base pressure with ion pump.

[d] Base pressure with oil diffusion pump.

[e] Oxidation failure of weld area.

[f] Complete wall penetration.

[g] Eight cycles total; 16 hours between four of the cycles and 4 hours between the other four cycles.

TABLE II. - ANALYSES OF T-111 AND DEPTH OF CONTAMINATION AFTER EXPOSURE AT 1260° C

[Exposure time, 96 hr.]

System pressure, torr	Analysis of T-111,[a] ppm				Depth of contamination,[b] mm	Oxygen content of lithium, ppm
	O	N	C	H		
---	36	30	22	0.6	---	110
Isothermal exposures						
[c] 1×10^{-8}	16	18	--	<0.5	0	320
[d] 5×10^{-8}	35	21	24	<0.5	0	250
5×10^{-7}	48	32	--	---	0	---
2×10^{-6}	105	37	--	---	.05	---
2×10^{-5}	726	75	42	0.6	.4	440
2×10^{-4}	3500	47	26	1.2	[e] 1.0	Very High
Cyclic exposure between 1260° and 540° C[f]						
[c] 1×10^{-8}	30	18	--	<0.5	0	200
2×10^{-5}	692	42	44	.8	.4	190
2×10^{-4}	3620	50	34	.8	[e] 1.0	53 000

[a] Oxygen and nitrogen determined by vacuum or inert-gas fusion. Carbon determined by combustion. Hydrogen determined by vacuum extraction.

[b] Determined by optical metallography and microhardness.

[c] Base pressure with ion pump.

[d] Base pressure with oil diffusion pump.

[e] Complete wall penetration.

[f] Eight cycles total; 16 hours between four cycles and 4 hours between the other four cycles.

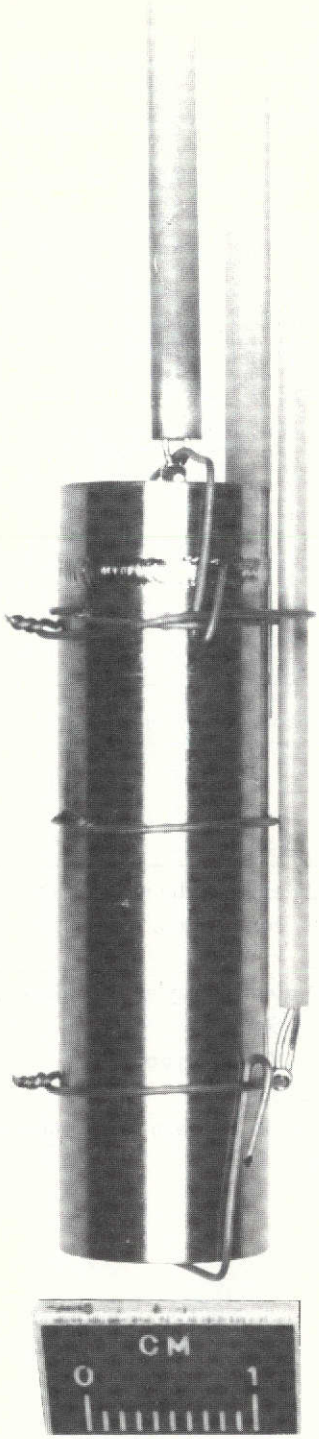

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Figure 1. - Capsule assembly.

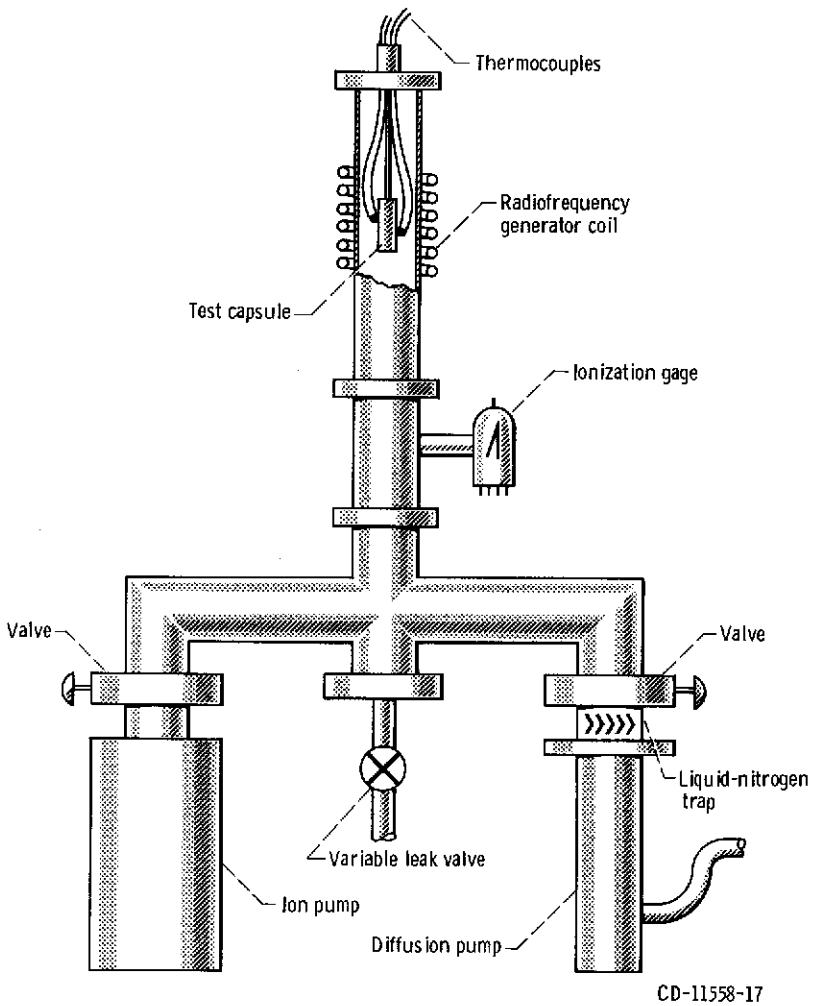


Figure 2. - Test apparatus.

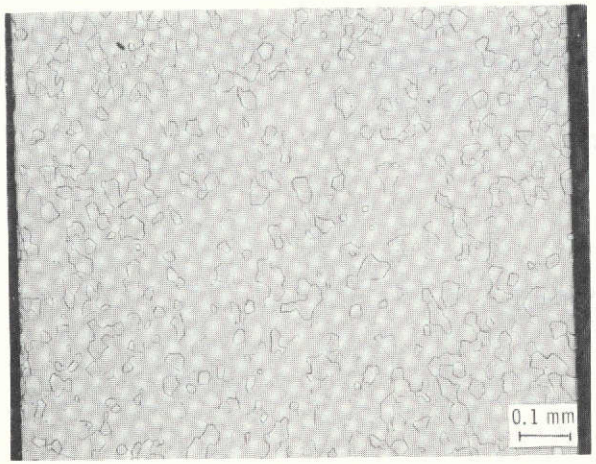
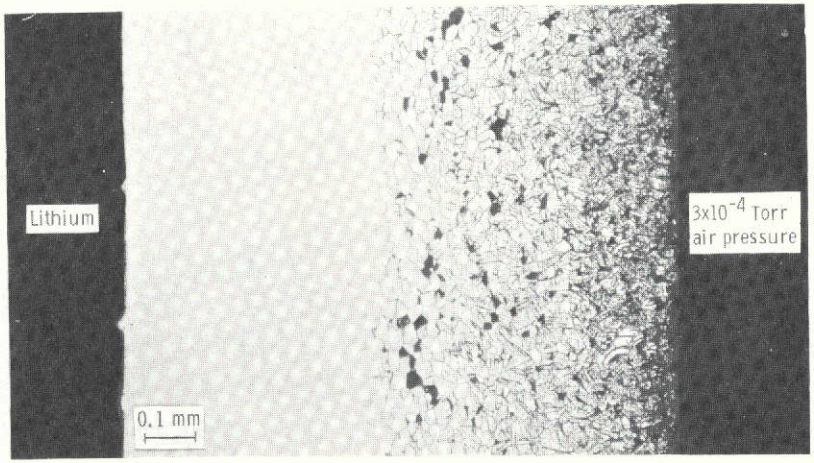
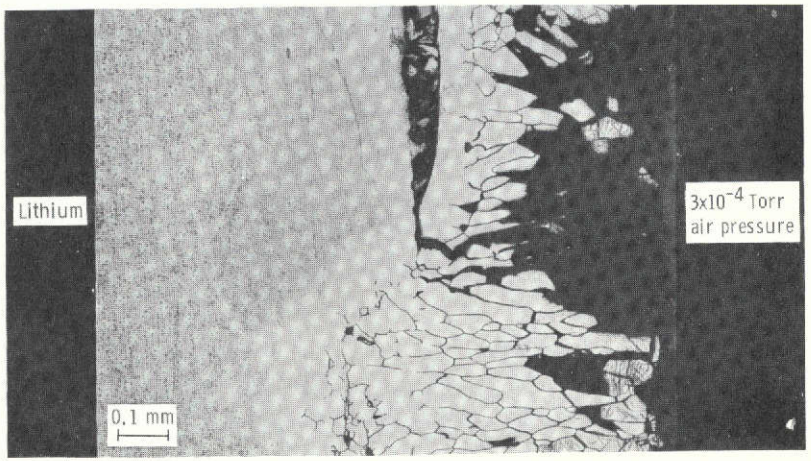


Figure 3. - T-111 annealed for 1 hour at 1316° C. Specimen etched. X100.



(a) Wall.



(b) Weld.

Figure 4. - T-111 isothermally tested at 980° C and 3x10^{-4} torr. Capsule failed after 18 hours. Specimen etched. X100.

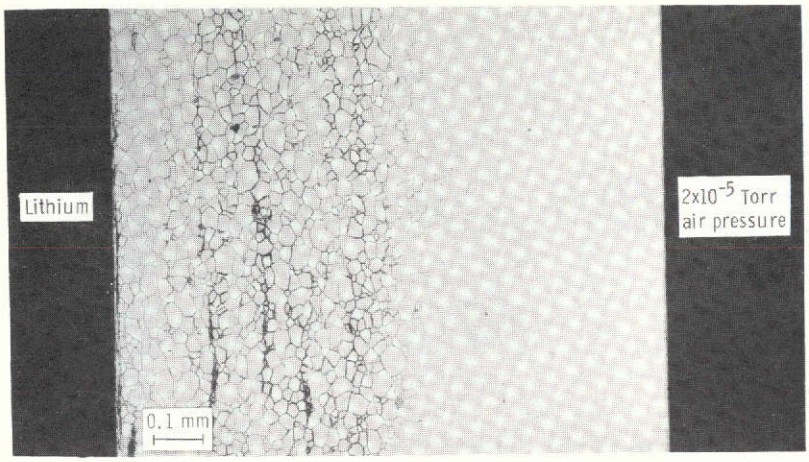


Figure 5. - T-111 cyclically tested at 980^{0} C and $2x10^{-5}$ torr for 96 hours. Specimen etched. X100.

Figure 6. - T-111 isothermally tested at 980^{0} C and $2x10^{-6}$ torr for 96 hours. Specimen etched. X100.

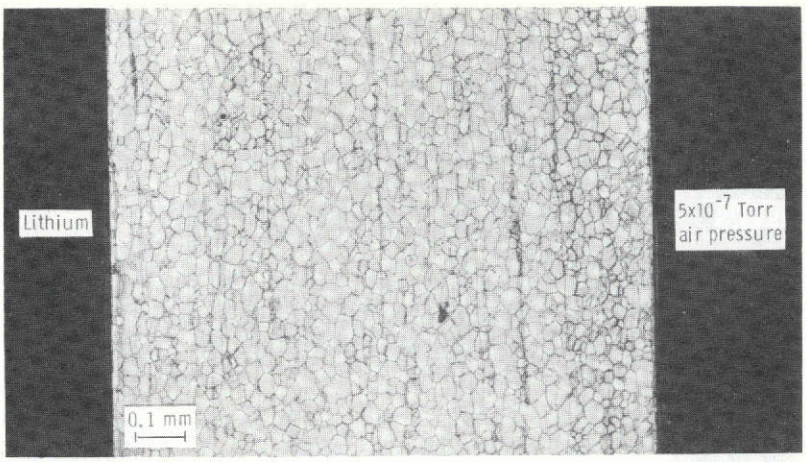


Figure 7. - T-111 isothermally tested at 980^{0} C and $5x10^{-7}$ torr for 96 hours. Specimen etched. X100.

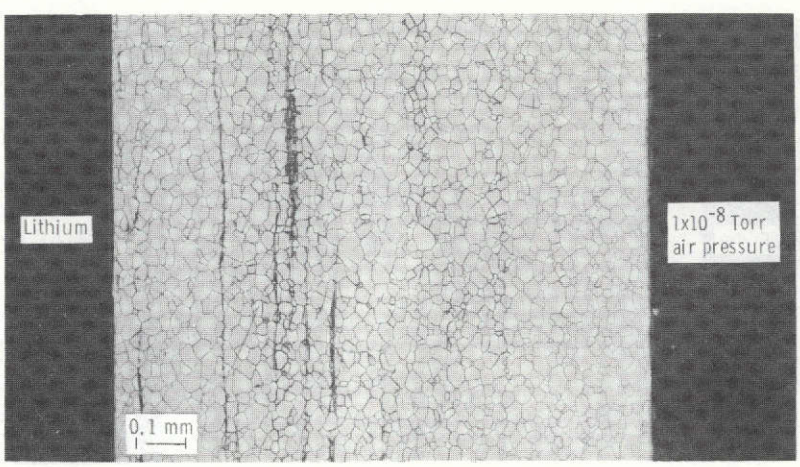
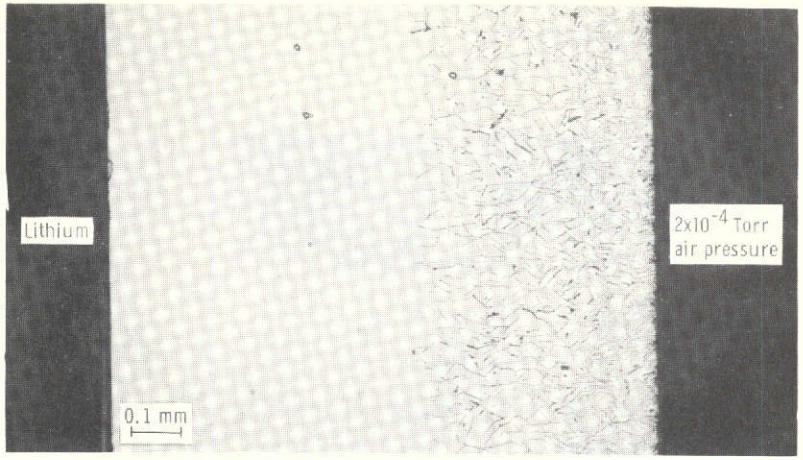
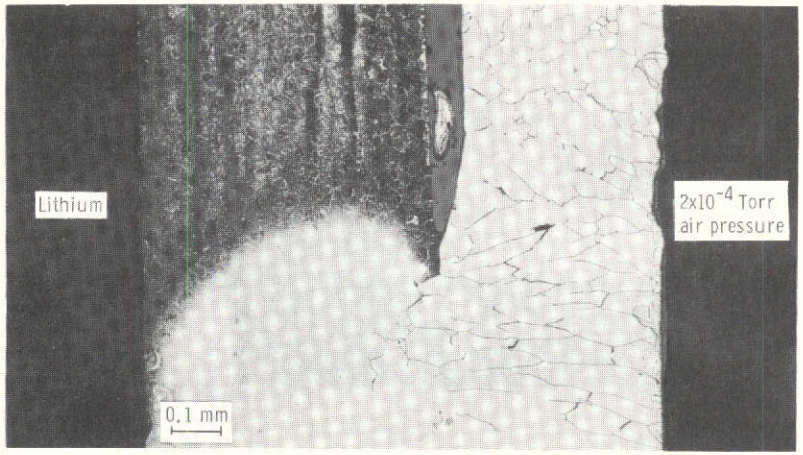


Figure 8. - T-111 isothermally tested at 980^{0} C and $1x10^{-8}$ torr for 96 hours. Specimen etched. X100.



(a) Wall.



(b) Weld.

Figure 9. - T-111 cyclically tested at 1260^{0} C and 2x10^{-4} torr for 96 hours. Specimen etched. X100.

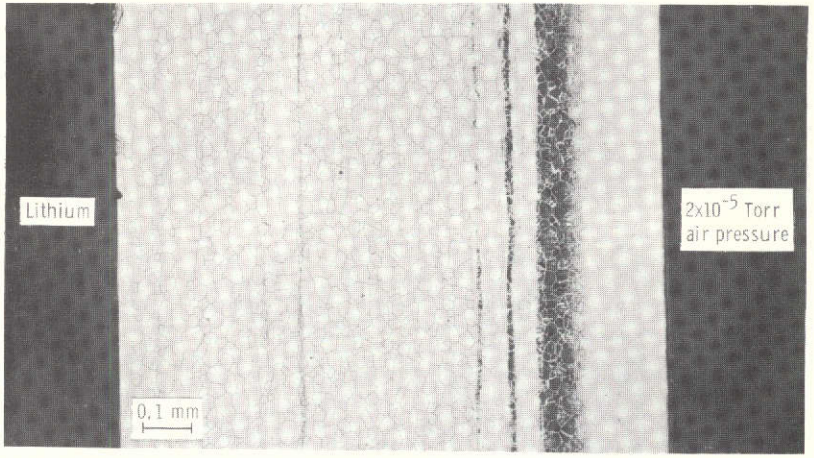


Figure 10. - T-111 cyclically tested at 1260^{0} C and 2x10^{-5} torr for 96 hours. Specimen etched. X100.

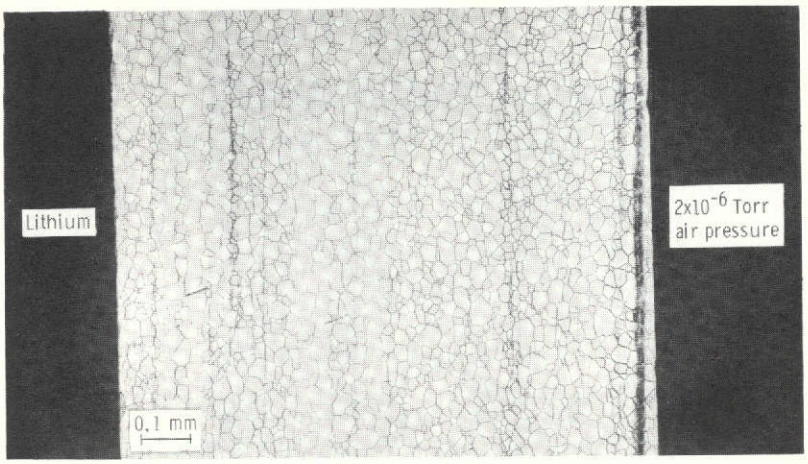


Figure 11. - T-111 isothermally tested at 1260^{0} C and 2x10^{-6} torr for 96 hours. Specimen etched. X100.

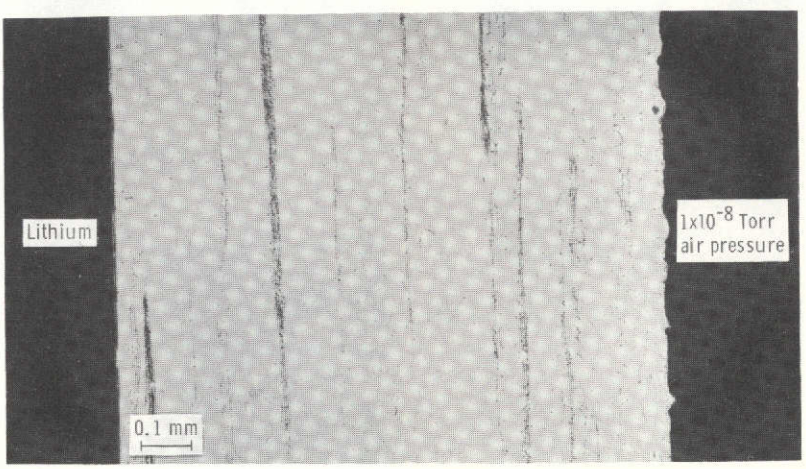


Figure 12. - T-111 isothermally tested at 1260^{0} C and 1x10^{-8} torr for 96 hours. Specimen etched. X100.

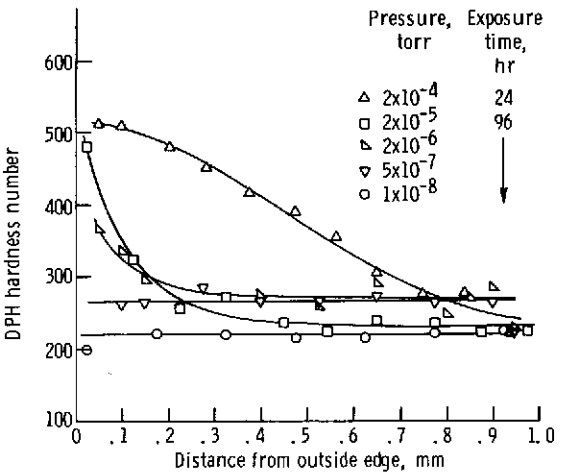


Figure 13. - Effect of pressure on wall hardness of T-111/ lithium capsules at 980° C.

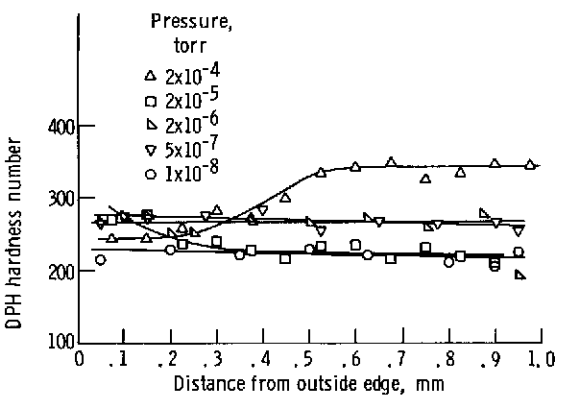


Figure 14. - Effect of pressure on wall hardness of T-111/ lithium capsules at 1260° C. Exposure time, 96 hours.